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PROCESS FOR RENDERING A TEXTILE MATERIAL RESISTANT TO MOISTURE

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The present invention relates to the treating of a textile material such as fabric or thread to intimately incorporate therein a dicarboxylic acid ester of cellulose and the product resulting therefrom.

Heretofore in the treating of textile fabric it has been necessary in coating or impregnating it with a cellulose ester to dissolve the ester in a volatile organic solvent which solution is then employed to apply the ester to the fabric. This process of treating fabrics has numerous disadvantages some of which are: expense of making up dopes or solutions of cellulose esters in volatile organic solvents, the danger of inflammability in dissolving the ester and in drying the cloth which has been treated with the solution, the necessity of recovering the solvent vapors to assure economy of operation and the care necessary to avoid stiffness or hardness in the fabric which has been treated.

One object of our invention is to provide a process for preparing a fabric impregnated with a cellulose ester and resistant to moisture in which the use of a solution of a cellulose ester in a volatile organic solvent is unnecessary. Another object of our invention is to provide a process of impregnating textile materials with a cellulose ester in which the danger of explosions or fires from solvent vapors is eliminated. Other objects will appear herein.

We have found that a textile material such as a fabric may be impregnated with a cellulose ester by treating that material with an aqueous solution of a water-soluble salt of a dicarboxylic acid ester of cellulose and then regenerating the ester therefrom by treating the material with a solution of an acid (either organic or mineral) which has an ionization constant greater than that of the second hydrogen of the dicarboxylic acid corresponding to the dicarboxylic radical which is joined to the cellulose; or in the case where the ammonium salt (or an amine salt which may be decomposed by heat) is employed in the treatment by subjecting the material to a nonscorching temperature at which that ammonium salt is unstable. The temperatures which are preferable to employ for this latter treatment lie within the range 60°–160° C. The speed with which the ammonium or similar salt is decomposed is dependent on the degree of temperature. For instance a temperature below 60° C. renders the decomposition too slow for high speed operation. However, where it is possible to subject the impregnated fabric to a raised temperature for a longer period temperatures below 60° C. may be employed. The salt may even be decomposed at room temperature, however a period of 5 or 6 hours or even more is necessary to remove substantially all of the basic constituent. At temperatures above 160° C. the ester will soften so that temperatures above 160° C. will not usually be employed unless that softening is unobjectionable. For instance if the process is carried out in such a manner that the ester will not adhere to the rolls or other parts of the apparatus if it is subjected to a softening temperature, obviously such a temperature may be employed.

If desired a plasticizer may also be incorporated in the cellulose ester by dissolving it in either the impregnating bath containing the salt of the cellulose ester or the acid bath for the precipitation of the ester or by spraying the plasticizer on the cloth as it passes thru the dryer or just prior to passing it thru calendering rolls. In the case where the plasticizer is added to the bath it is preferable to employ a water soluble plasticizer such as diethylene glycol, triethylene glycol or diethylene glycol diacetate rather than modifying the bath to render it soluble of the plasticizer which it is desired to employ. A salt of a mono ester of phthalic acid can also be employed as plasticizer. This salt is water soluble and can be incorporated in the water solution of the salt of the dicarboxylic acid ester of cellulose. During the subsequent acid treatment the water insoluble mono-ester is regenerated. If the plasticizer is to be incorporated in the ester by spraying it upon the cloth any of the ordinary liquid plasticizers such as dimethyl phthalate, diethyl phthalate etc. may be employed.

The following examples are illustrative of the carrying out of the present invention:

Example I

15 lbs. of the sodium salt of cellulose acetate phthalate is dissolved in 10 gallons of water and 4 yards of a loosely woven unsized cotton cloth is steeped therein until completely wet with the solution. The cloth is then allowed to drain and is passed into aqueous acetic acid (10%) whereupon the cellulose acetate phthalate is regenerated and this compound is intimately associated with the fabric. The cloth is then dried and is passed thru calendering rolls at a temperature of 60°–160° C., preferably 105° C., resulting in a cloth having resistance to the passage of moisture.

Example II

5 lbs. of the ammonium salt of cellulose ace-

tate phthalate is dissolved in 2 gallons of water and 1 lb. of diethylene glycol is added to plasticize the ester formed. 2 yards of unsized cloth is soaked therein until the entire cloth has been wet by the solution after which the cloth is drained and dried. The cloth is then passed thru calendering rolls at a temperature of about 105° C. which treatment liberates the ammonia and regenerates the cellulose acetate phthalate.

The preparation of the water soluble salts of the dicarboxylic acid esters of cellulose is disclosed and claimed in Malm and Waring applications Serial Nos. 380,252 filed July 22, 1929, 627,149 filed July 30, 1932 and in Fordyce Patent No. 1,969,741. For instance the sodium salt of cellulose acetate phthalate may be prepared by dissolving the acetate phthalate in ethylene glycol monomethyl ether and adding thereto a solution of sodium hydroxide in a mixture of equal volumes of water and ethylene glycol monomethyl ether until the point of neutrality is reached. The ammonium salt may be prepared by dissolving the cellulose acetate phthalate in anhydrous acetone and then introducing a current of dry ammonia gas therein. The pyridine salt is formed by esterifying a cellulosic material with a dicarboxylic acid anhydride in the presence of pyridine.

As was pointed out before the cloth treated in accordance with the present invention exhibits the characteristic of resistance to the passage of moisture so that in addition to the ordinary uses to which it might be put it is also eminently suitable for making sugar sacks or in fact containers for any material which is affected either by the loss or the addition of moisture.

Various water-soluble salts of the dicarboxylic acid esters of cellulose are suitable for use in a process embodying the present invention some of which are:

Cellulose acetate sodium phthalate
Cellulose acetate ammonium phthalate
Cellulose acetate potassium phthalate
Cellulose acetate pyridine phthalate
Cellulose sodium phthalate
Cellulose ammonium phthalate
Cellulose triethanolamine phthalate
Cellulose potassium phthalate
Cellulose acetate sodium succinate
Cellulose acetate ammonium succinate
Cellulose acetate potassium succinate
Cellulose sodium succinate
Cellulose ammonium succinate
Cellulose potassium succinate
Cellulose acetate sodium diglycolate

In carrying out the present process the aqueous solution of the salt may be applied to the textile material by steeping, by a coating blade or by any of the well known means of treating fabrics. For continuous operation it is desirable to provide rolls for continuously passing the cloth thru the respective baths and leading it thru the drying and calendering means.

Instead of using acetic acid in aqueous solution for removing the basic constituent of the salt, other acids or other solutions might be employed. For instance an aqueous solution of a mineral acid such as sulfuric, hydrochloric or phosphoric or of some strong organic acid such as formic might be employed in this connection.

Other solvents such as alcohol (ethyl or methyl) or ethyl ether might be employed as the diluent of the acid in this neutralization step, the only criterion being that the liquid be a solvent for the acid employed and substantially a non-solvent of the cellulose ester resulting from the acid treatment.

It is also to be understood that other solvents than water which are similar in character such as an alcohol (ethyl or methyl) might be employed as the solvent for the water-soluble salt, which solution is employed to impregnate the textile material.

We claim as our invention:

1. A process for rendering a textile material resistant to moisture which comprises thoroughly wetting the material with a substantially neutral aqueous solution of a water-soluble salt of a cellulose ester containing a dicarboxylic acid radical and then regenerating the cellulose ester.

2. A process for rendering a textile material resistant to moisture which comprises thoroughly wetting the material with a substantially neutral aqueous solution of a water-soluble salt of a cellulose ester containing a dicarboxylic acid radical and then regenerating the cellulose ester by means of a dilute acid having an ionization constant greater than that of the second hydrogen of the dicarboxylic acid corresponding to the radical present in the ester.

3. A process for rendering a textile material resistant to moisture which comprises thoroughly wetting the material with a substantially neutral aqueous solution of an ammonium salt of a cellulose ester containing a dicarboxylic acid radical and then regenerating the cellulose ester by subjecting the material to a non-scorching temperature of at least 105° C.

4. A process for rendering a textile material resistant to moisture which comprises thoroughly wetting the material with a substantially neutral aqueous solution of a water-soluble salt of a cellulose ester containing phthalyl groups and then regenerating the cellulose ester.

5. A process for rendering a textile material resistant to moisture which comprises thoroughly wetting the material with a substantially neutral aqueous solution of a water-soluble salt of a cellulose ester containing phthalyl groups and then regenerating the cellulose ester by means of acetic acid.

6. A process for rendering a textile material resistant to moisture which comprises thoroughly wetting the material with a substantially neutral aqueous solution of a water-soluble salt of a cellulose ester containing a dicarboxylic acid radical containing a water-soluble plasticizing material and then regenerating the cellulose ester.

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